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THERMAL ANALYSIS SYSTEM

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Final Report for
Contract No. AFOSR 87-0022
submitted to
Air Force Office of Scientific Research
Building 410
ATTN: Dr. A. Rosenstein
Bolling Air Force Base
Washington, DC 20332

Submitted by

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The Perkin Elmer Thermal Analysis System Consists of various individual units like the Differential Thermal Analyzer (DTA)., Differential Scanning Calorimeter (DSC), Thermogravimetric Analyzer (TGA) and the Thermomechanical Analyzer (TMA). Two controllers, two dedicate computers, one graphics plotter and one microbalance make up the entire package. The system was installed in November 1986 and was first put into operation the early part of 1987.

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THERMAL ANALYSIS SYSTEM

The Perkin Elmer Thermal Analysis System consists of various individual units like the Differential Thermal Analyzer (DTA), Differential Scanning Calorimeter (DSC), Thermogravimetric Analyzer (TGA) and the Thermomechanical Analyzer (TMA). Two controllers, two dedicated computers, one graphics plotter and one microbalance make up the entire package. The system was installed in November 1986 and was first put into operation during the early part of 1987.

A brief description of each of the units used so far and the results obtained follows.

DTA and DSC

The DTA 1700 system measures the difference in temperature between the sample under study and an inert reference sample (i.e. a sample which does not undergo any phase transition, either solid to solid or solid to liquid) when the temperature of the furnace is programmed at an operator selected heating or cooling rate. The normal operating temperature range for this instrument is from ambient to 1500°C. The range of heating rate is 0.2°C/min to 100°C/min. Argon is used at a flow rate of 40-45 cc/min to provide an inert atmosphere. If the sample under study undergoes a phase transformation, there is an associated heat liberation or heat absorption by the sample, depending on whether the phase transformation is exothermic or endothermic. This heat or energy imbalance is manifested as temperature rise or temperature decrease of the sample under study in comparison to the reference. This difference in temperature is plotted as a function of the furnace temperature to indicate precise phase transformation temperatures.

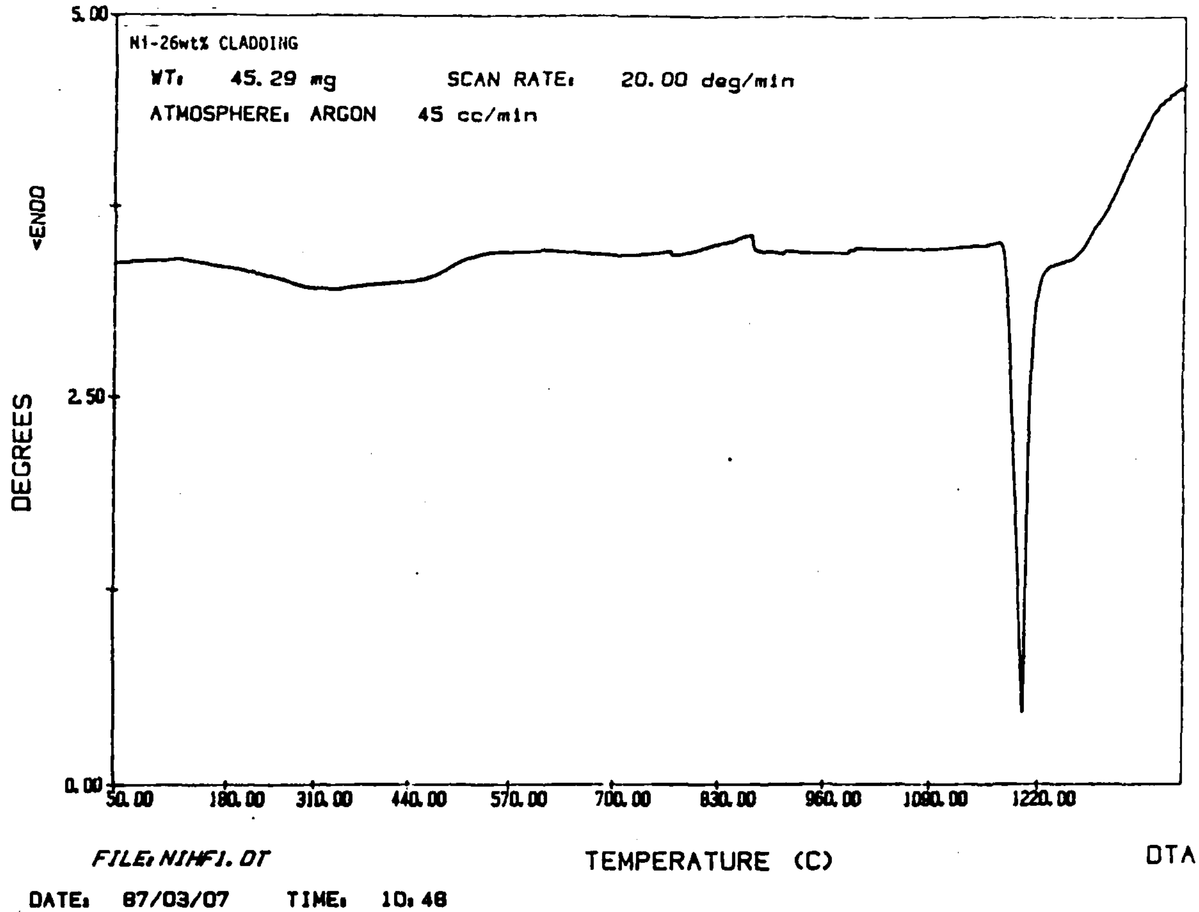

Figure 1(a) DTA plot of Ni-26wt% Hf cladding indicating eutectic transformation

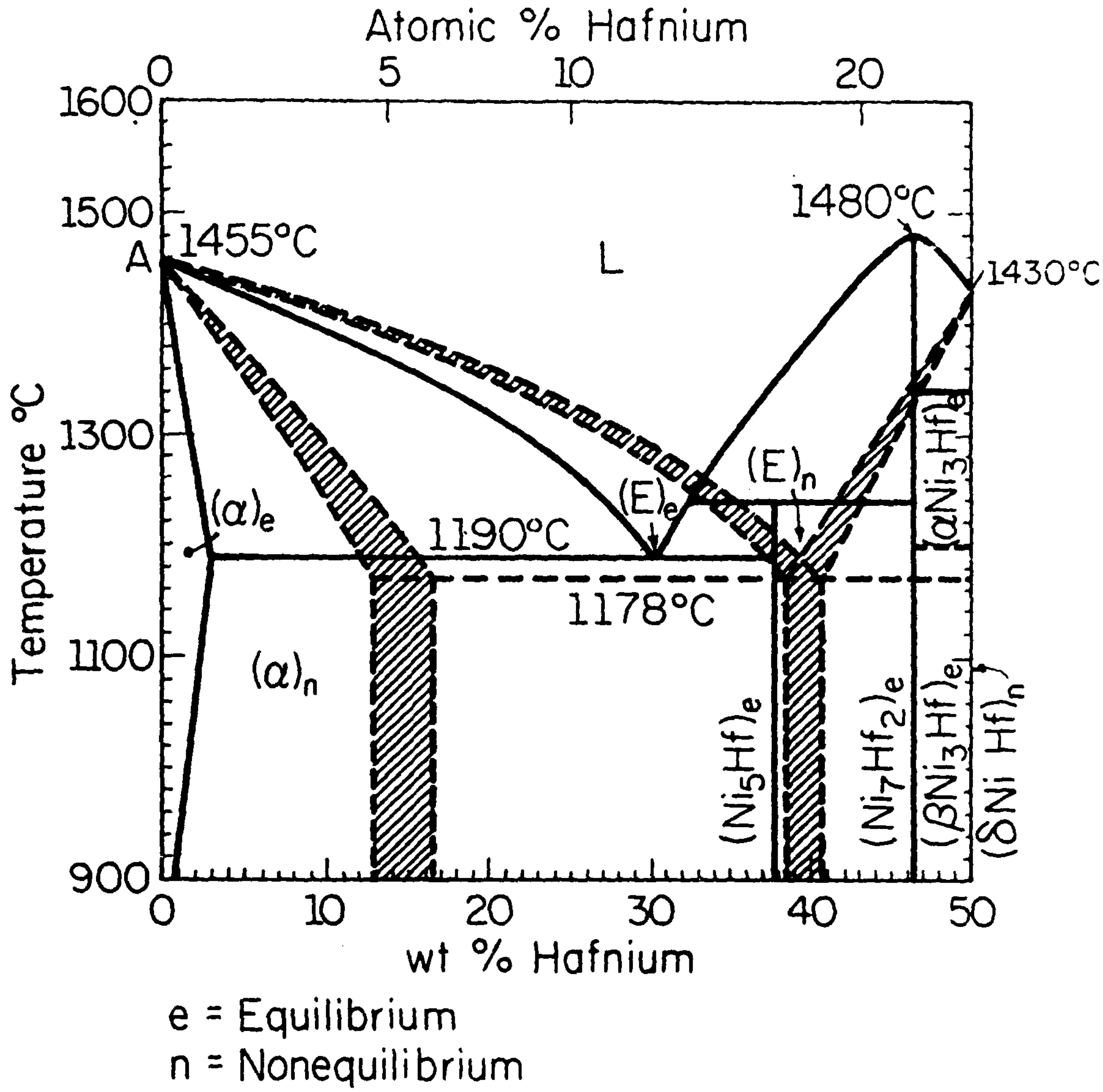

Figure 1(b) Equilibrium and non-equilibrium (dashed lines) plot of the Ni-Hf binary phase diagram

In the case of the DSC, however the operation range of temperatures is from -170°C to 725°C. The heat/energy liberated or absorbed by the sample is measured by a calorimeter and the resultant output is plotted as a function of the furnace temperature. Hence, thermodynamic quantities like heat of transition, entropy of transformation etc., which are related to the phase transition can be obtained very precisely using a DSC.

The DTA and the DSC have been used quite frequently during the course of investigation under the Air Force research program.

(a) The quick cooling rate associated with the laser cladding process gives rise to a number of novel nonequilibrium, metastable, phases which extend very attractive properties to the claddings. However, in order to understand the stability of these phases at elevated temperatures, the kinetics and thermodynamics of the transformation of these phases to equilibrium products and also to characterize these phases distinctly by their thermal effects the DTA and DSC are used. The results obtained via using these two techniques also help us to ascertain the degree of deviation that is obtained in phase transformation temperatures due to nonequilibrium cooling rates associated with laser cladding process.

(b) Ni-Hf binary nonequilibrium phase diagram was partially resolved (eutectic transformation temperature) using the DTA. (Fig. 1(a) and (b)).

(c) The shift in the Nb-Ti (Nb rich end) phase diagram is being determined now via DTA techniques. (Fig. 2).

(d) The γ' dissolution temperature of both the claddings and the substrate (Rene 80) was determined in order to get an idea of the temperature up to which the materials have comparable physical properties. (Fig. 3).

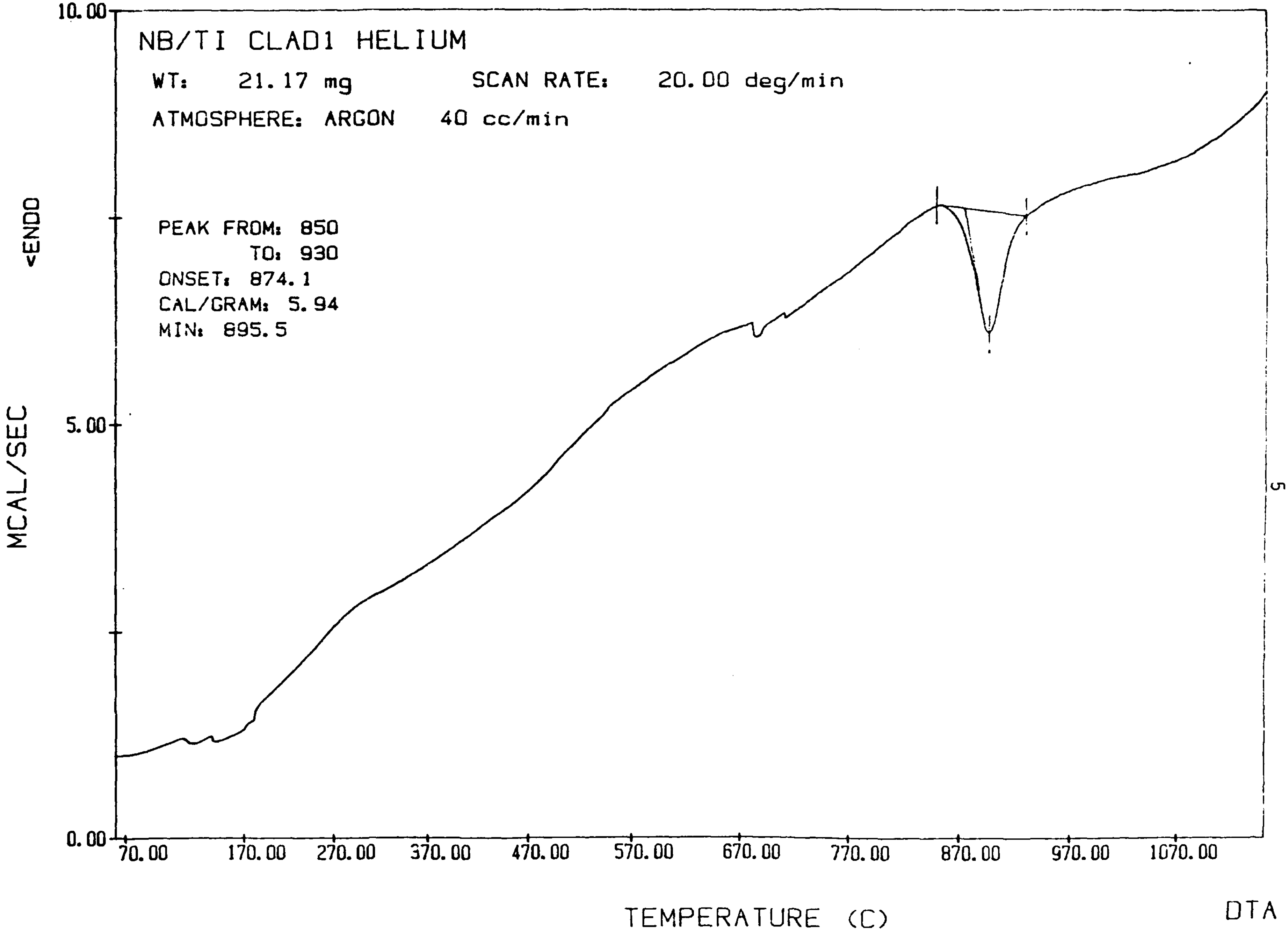

Figure 2 DTA plot of Ti-Nb $\alpha \rightarrow \beta$ transition

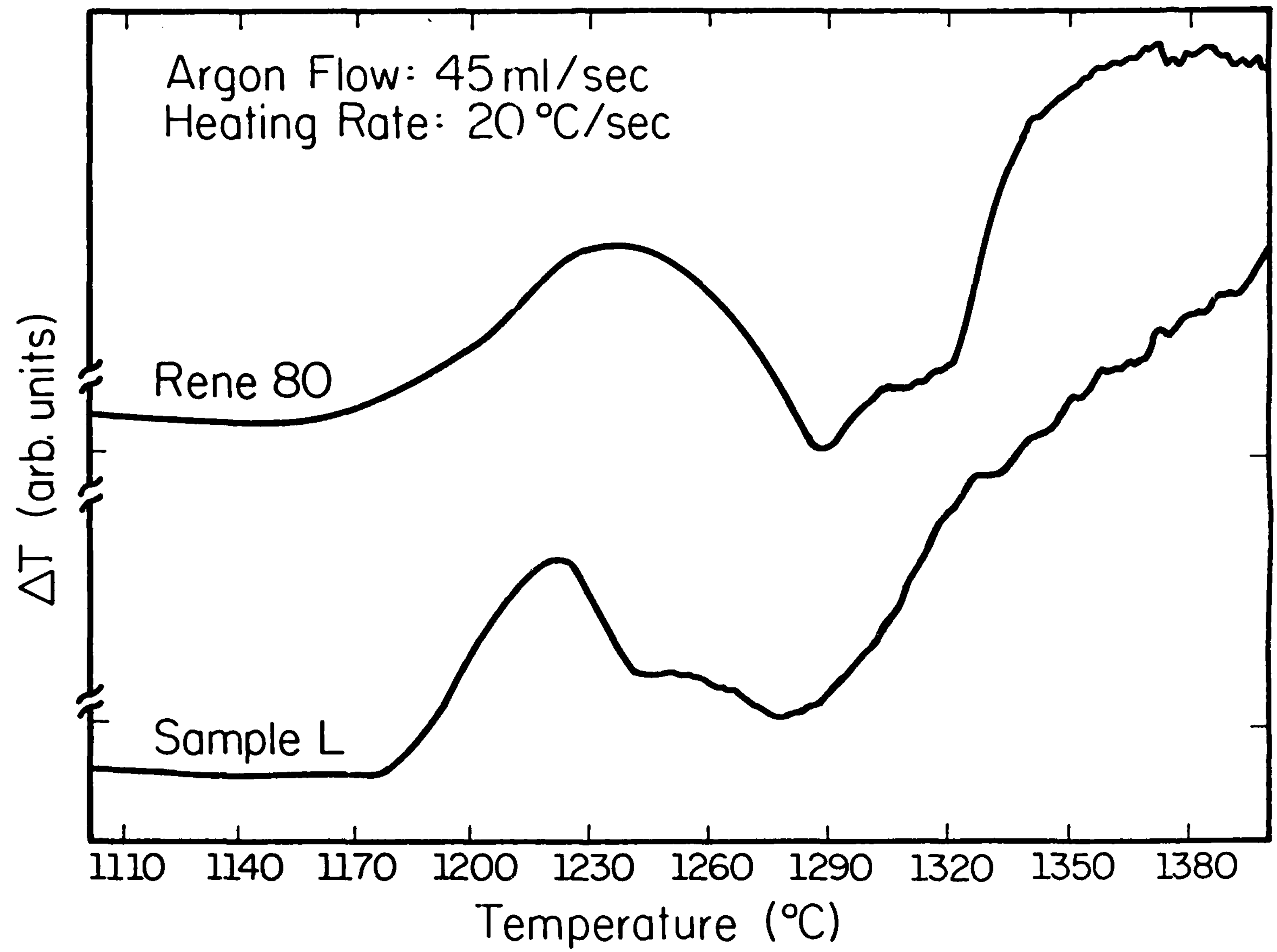

Figure 3 γ' dissolution temperature study using DTA for both Rene 80 (substrate) and cladding (NiAlCrHf) sample

(e) Possibility of forming metastable phases and amorphous phases in magnesium systems is also being studied. If an amorphous phase forms during cladding, then, on reheating, a crystallization point can be observed when this amorphous phase becomes crystalline. At present various alloying additions are being studied to see this effect. It must be noted that the impetus of forming an amorphous phase is to produce a cladding so as to improve the corrosion properties of magnesium and its alloys.

TGA

The Perkin-Elmer TGA-7 was used to measure the oxidation resistance of the Ni-Al-Cr-Hf claddings and the substrate. This system features both computerized system control and data acquisition capability (Fig. 4). The change in the mass of the sample is measured dynamically as a function of oxidation time. The analyzer itself during operation consists of a furnace surrounding a platinum sample pan suspended from a microbalance by a platinum wire. Samples are stood up on their width and held in place between two platinum retaining wires. 0.3 ml/s of dry air flows past the sample and 0.6 ml/s of Ar is used to protect the microbalance during operation. The sample is heated from ambient at 100°C/min to 1200°C and held at 1200°C for 8 hours. In situ cooling is then performed at a rate of 10°C/min to 950°C before cooling to ambient at a rate of 25°C/min.

The samples are sectioned from the cladding using a low speed saw with a SiN blade (Fig. 5). Following sectioning, all of the faces of the sample are progressively abraded through 600 grit using SiC paper with water as the lubricant. The dimensions of the samples are measured with caliper and micrometer prior to a final cleaning with (1) acetone and (2) trichloroethane.

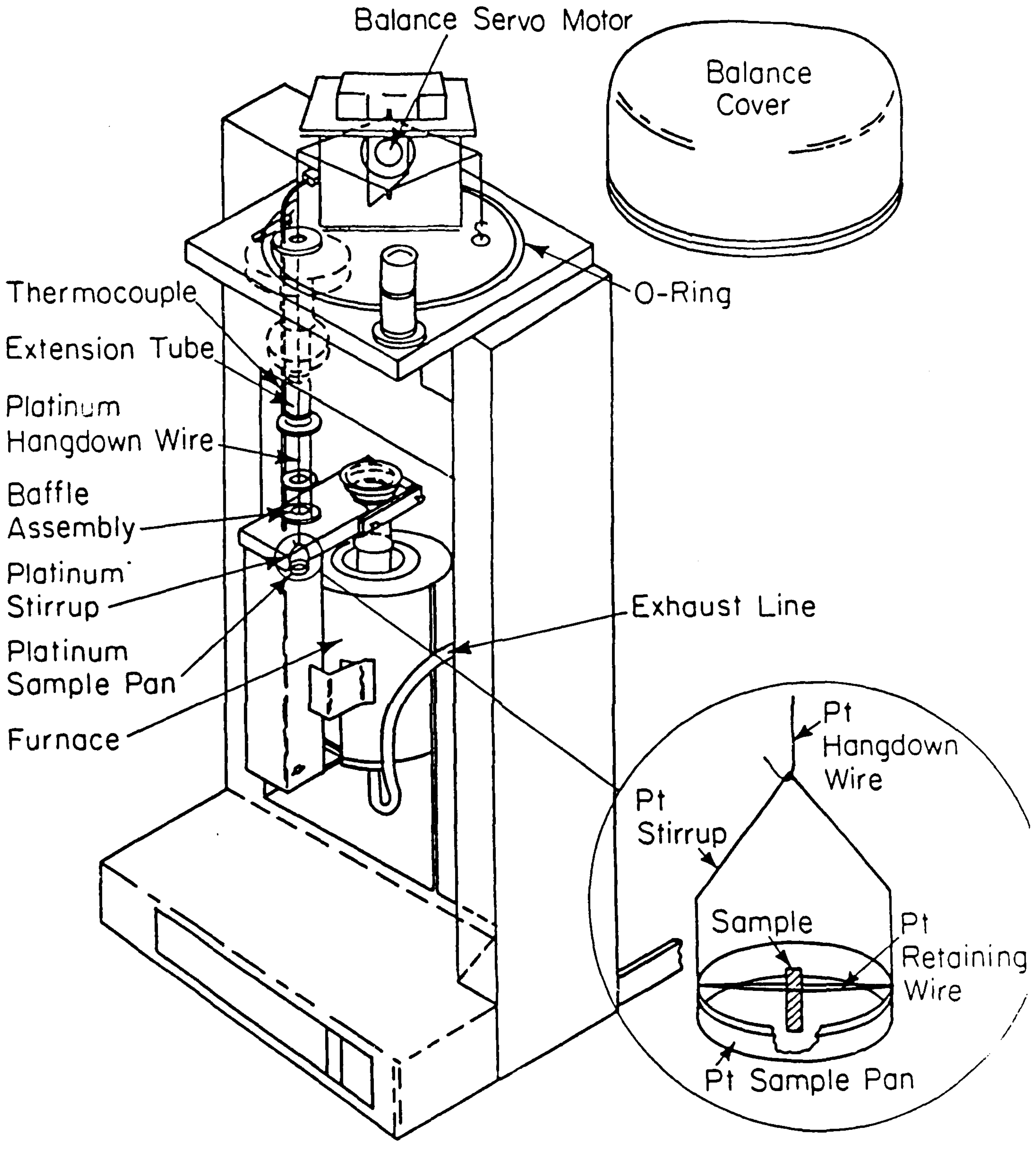

Figure 4 TGA-7 schematic (Furnace Positioned for clarify)

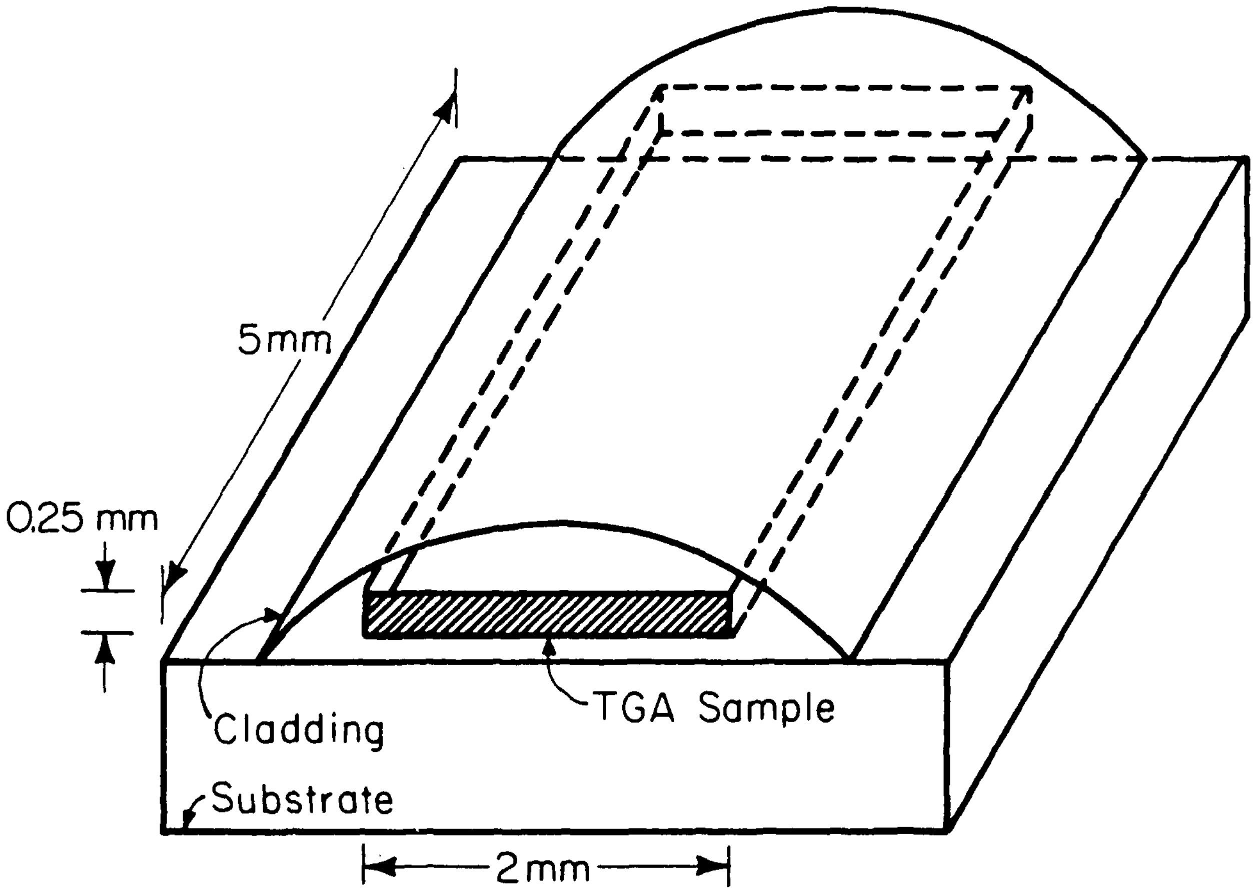

Figure 5 Location of the oxidation test samples within the laser claddings

Some variation in the sample dimensions was required to test all of the claddings.

A plot of the sample mass per unit sample area as a function of oxidizing time for the claddings is shown in the attached figure (Fig. 6). Samples J and L2 were sectioned from claddings of the above composition. Both of the cladding samples gained significantly less weight than the substrate (mean of two samples). This indicates that the claddings have oxidation resistance superior to that for the substrate. Examination of the cross-section of the samples indicated that the claddings also lost less metal to oxidation than the substrate samples.

Other compositions are also being studied in both Ni-based superalloy claddings and in Nb-based alloys. The TGA is being used to constantly monitor the oxidation properties of the claddings and optimizing the cladding compositions.

In summary, this instrument has been extremely useful in studying the high temperature properties of metastable materials synthesized by laser cladding, which is the primary goal of the AFOSR 85-0533 project. Already six papers are either published or awaiting publication which utilized the data gathered using this system. These papers are listed in Appendix 1.

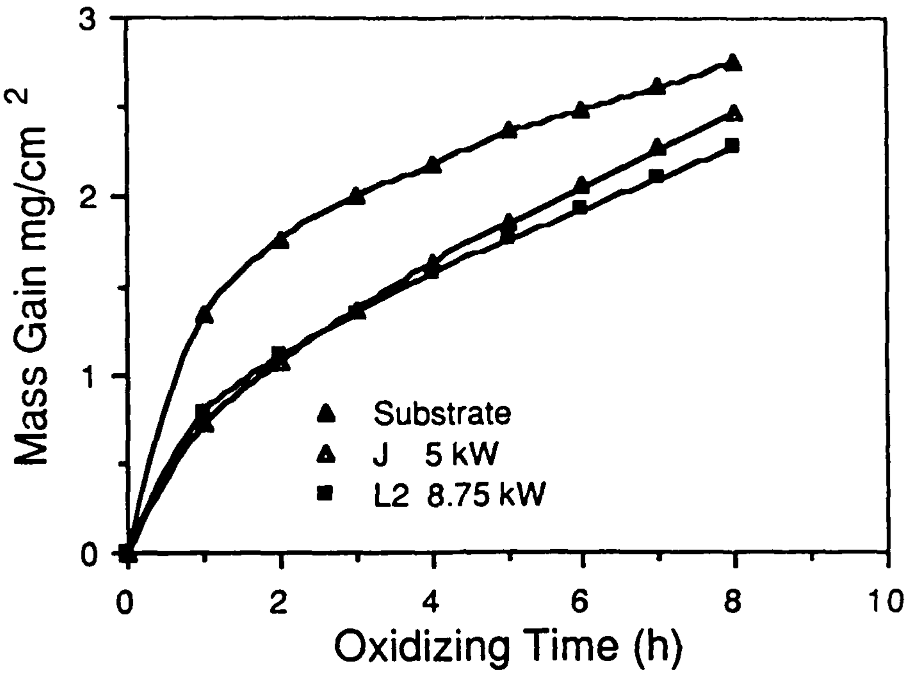

Figure 6 Oxidation mass gain/unit area of laser claddings in air at 1200°C as a function of exposure time

APPENDIX 1

PUBLICATIONS LIST FOR THE AFOSR GRANT NO. AFOSR 5-0333

1. J. Singh, K. Nagrathnam and J. Mazumder, "Laser Cladding of Ni-Cr-Al-Hf on Inconel 718 for Improved High Temperature Oxidation Resistance," High Temp. Technology Vol. 5, No. 3, pp. 131-136, August, 1987.

2. S. Sircar, C. Ribaudo and J. Mazumder, "Laser Clad $Ni_{70}Al_{20}Cr_{7}Hf_{3}$ Alloys with extended Solid Solution of Hf: Part I: Microstructure Evolution," submitted to Met. Trans. A.

3. S. Sircar, C. Ribaudo, A. Kar, and J. Mazumder, "Microstructure, Oxidation Properties and Nonequilibrium Solubility in Laser Clad Ni-Based Alloys," Invited Paper, Proc., AWS Surface Modification Conf., New Orleans, April 14-16, 1988.

4. J. Mazumder, S. Sircar, C. Ribaudo, and A. Kar, "Microstructure and Oxidation Properties of Laser Clad $Ni_{70}Al_{20}Cr_{7}Hf_{3}$ Alloys with Extended Solid Solution of Hf," Invited Paper, Proc., SPIE Lasers in Manufacturing Conf., Portugal, June 2-5, 1988.

5. C. Ribaudo, S. Sircar, and J. Mazumder, "Laser Clad $Ni_{70}Al_{20}Cr_{7}Hf_{3}$ Alloys with Extended Solid Solution of Hf: Part II, Oxidation Behavior," submitted to Met. Trans. A.

6. S. Sircar, J. Singh and J. Mazumder, "Microstructure and Extended Solid Solubility of Hf in Ni-26 wt%Hf Binary Alloy by Laser Cladding," submitted to Acta. Met.